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DEVELOPMENT OF 400°F SEALANTS FOR FLAT PLATE SOLAR COLLECTOR
CONSTRUCTION AND INSTALLATION

Final Report, October 1, 1978–September 30, 1979

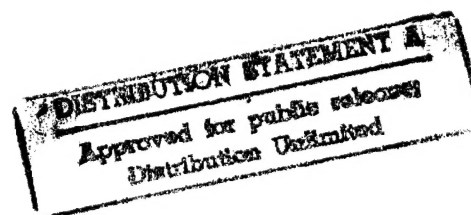
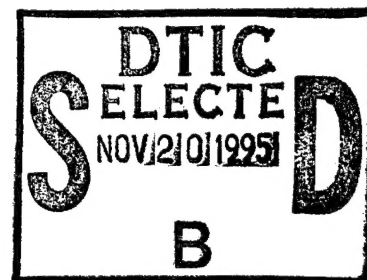
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DEVELOPMENT OF 400°F SEALANTS
FOR FLAT PLATE SOLAR COLLECTOR CONSTRUCTION AND INSTALLATION

FINAL REPORT
FOR THE PERIOD
1 OCTOBER 1978 - 30 SEPTEMBER 1979

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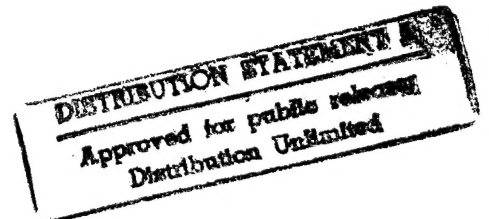


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ABSTRACT

Twenty candidate sealants representing ten different polymer types were evaluated as potential solar collector sealants. Polymer types tested included epichlorohydrin rubber, EPDM rubber, silicone, polysulfide, acrylate rubber, and a fluoroelastomer.

Initial screening of sealants consisted of measuring high temperature stability and adhesion retention.

Several sealant compositions exhibited satisfactory performance in these tests and were selected for further evaluation. These materials were based on an EPDM rubber, a Viton® fluoroelastomer, and silicone polymers.

Further testing of these candidate materials included determination of adhesion retention under UV/water/heat conditions, fogging temperature, low temperature flexibility, and physical properties. Four silicone-based materials appeared to be suitable candidates for sealing solar collectors. These include Dow Corning 90-006-02 and 3120, General Electric 1200, and PR-1939 from Products Research and Chemical Corporation.

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FINAL REPORT

CONTRACT NO. DE-AC04-78CS35303

DEVELOPMENT OF 400°F RESISTANT SEALANTS FOR FLAT PLATE SOLAR COLLECTOR CONSTRUCTION AND INSTALLATION

INTRODUCTION

The purpose of this contract was to screen, select, and develop sealant formulations for use in flat plate solar collector construction and installation. Toward this end, ten different polymer types, including epichlorohydrin rubber, acrylate rubber, acrylonitrile rubber, thiol-terminated polyoxypropylene, a proprietary polysulfide, a silane-terminated polyoxypropylene, an aliphatic epoxy, several silicones, and an EPDM rubber were evaluated in sealant compositions. Additionally, a number of commercially available sealants were to be evaluated as potential solar collector sealants.

Several products or polymers which were not mentioned in the original proposal have been included in this study, while several which were mentioned have been excluded. Generally, this was necessary because of current product availability or to avoid repetition in testing two very similar materials.

Flat plate solar collectors can operate at 200°F during the daylight hours and cool off to subfreezing temperatures at night. Temperatures may rise to over 400°F if flow of the heat exchanging fluid is interrupted or if concentrating mirrors and lenses are used in the design. This wide operating

temperature range requires that any potential solar collector sealant be elastomeric under these temperature extremes. In addition, the sealant must have a sufficiently low modulus to accomodate the stresses which are produced as a result of differences in linear thermal expansion of such dissimilar materials as aluminum and glass. Not only must the sealant permit free movement of the unit to avoid excessive stress on glass lites, but it must maintain adhesion under all conditions of stress, exposure to ultraviolet radiation (UV), high temperature, low temperature, 100% relative humidity, and dry heat. Additionally, the sealing material must be non-fogging if initial transparency of the unit is to be maintained. Fog testing of candidate sealants has been performed according to the Architectural Aluminum Manufacturer's Association (AAMA) specification.

Adhesion of each candidate sealant was evaluated on three common solar collector construction materials: glass, anodized aluminum and galvanized steel.

Test specimens were exposed to combinations of heat, humidity and ultraviolet light in an effort to simulate a solar collector environment.

All glass adhesion panels were tested at 140°F to 160°F. Test specimens were made by applying the sealant to the test panels in a 30 mil thickness (± 10 mils) and curing for seven days at ambient temperature. The panels were placed sealant side down on cellulose sponges which were continuously floating in distilled water. Ultraviolet lamps were placed 18 inches above the specimens.

Direct UV exposure of the sealant bond line was obtained by leaving the

glass uncovered in some specimens. In other specimens, the glass was covered with aluminum foil or masking tape to obtain varying degrees of UV exposure.

On the sealant side, all specimens were covered with aluminum foil on half of the exposed sealant area to examine the effect of the exclusion of water and atmospheric gases.

Thus, in the case of glass, the following six environments were created; UV/with water, diffused UV/with water, no UV/with water, UV/no water, diffused UV/no water, and no UV/no water.

When using metal test substrates, only two conditions were tested since the bond line could not be exposed directly to UV. Exposure conditions were no UV/with water and no UV/without water. In the tests, oxygen always accompanies water exposure.

To simulate cyclic stresses to which a sealant is subjected, potential sealants were tested to ASTM C719. This specification, which is a test used in the construction industry, subjects a sealant to cyclic stress such as would be encountered in a typical building joint. In addition the test procedure was modified to test specimens at 200°F both in dry heat and in 100% RH instead of ambient conditions.

However, laboratory tests do not predict sealant behavior in the field with complete accuracy. To duplicate field conditions as closely as possible, simulated solar collectors have been constructed and exposed using PR-1939.

PHASE I

PRELIMINARY HIGH TEMPERATURE TESTING

Commercial sealant suppliers were solicited to supply samples of their sealants for evaluation as potential solar collector materials. Seven products were received and evaluated: all of them are based on silicone polymers. These were Dow Corning's 3120, 90-006-02, 795, 732-CL-11, X-3-7058, and General Electric's 1200 and Silpruf.

In order to provide a rapid means of evaluating potential sealant candidates, the initial stage of this contract was concerned with high temperature testing of the candidates. Temperatures chosen were 302°F (150°C), 392°F (200°C), and 437°F (225°C). Tensile strength, tear strength and percent elongation were obtained after an ambient and/or heat cure, and after heat aging. This testing was very effective in quickly eliminating a large percentage of potential sealants. Table I is a general summary of these tests, while Table II summarizes all first stage testing and gives all data.

The following materials were tested under Phase I:

Sylgard 184

This commercial product from Dow Corning is a platinum-catalyzed silicone polymer-based material. Cured test specimens were prepared as follows: Sheets of silicone release paper were placed inside a steel mold. After pouring the catalyzed, degassed silicone onto the paper, the sample was pressed to a thickness of 75 to 80 mils. This sandwich was then removed from the mold and allowed to cure overnight at ambient temperature. After removal of the partially cured silicone sheet from the release paper, the material was cured for 16 hours at 120°F, followed by 16 hours at 180°F.

Tensile and tear specimens were then cut from the cured sheets according to ASTM D412. Four sets of specimens were made, one for initial physical properties and one for each of the three test temperatures. As shown in Table II, initial tensile strength of this material was 940 psi, tear strength was 50 pli, ultimate percent elongation was 140%, and hardness was 45 Shore A.

Physical properties after two weeks at 302°F were 937 psi tensile strength, 60 pli tear strength, and 126% ultimate elongation. This represented only a minor change, almost within experimental error. Above 302°F, physical properties fell off drastically after two weeks' exposure.

After only one week at 392°F, initial values had decreased to 220 psi tensile strength, 48 pli tear strength, and 80% ultimate elongation. While tensile strength fell significantly, tear strength and ultimate elongation remained relatively constant. After two weeks at 392°F, these properties were 189 psi tensile strength, 50 pli tear strength, and 50% ultimate elongation.

Results for 437°F testing were similar to those at 392°F. One week aging gave 220 psi tensile strength, 48 pli tear strength, and 80% ultimate elongation while these values after two weeks were 199 psi tensile strength, 43 pli tear strength and 50% ultimate elongation.

Visual examination of the test specimens during oven aging showed no changes at the lower test temperature of 302°F, but after 74 hours, those specimens exposed at higher temperatures exhibited a gradual darkening.

This material was dropped from further consideration due to large decreases in tensile strength after heat aging.

PR-1977

This is a silicone-based product which is commercially available from

Products Research & Chemical Corporation. Though overall test results for this material were satisfactory, there was a steady decline in percent elongation with continued heat exposure.

Test specimens were made according to ASTM D412. This product will not cure well in a closed mold, therefore the same technique was used to cast a sheet as described for Sylgard 184.

Initial physical properties were 660 psi tensile strength, 65 pli tear strength, and 60% ultimate elongation. After one week at 302°F, these values changed to 605 psi tensile strength, 51 pli tear strength, and 56% ultimate elongation respectively. No further decline was noted after another week at 302°F and the values were 615 psi tensile strength, 58 pli tear strength, and 53% ultimate elongation. Hardness initially was 73 Shore A. After the first week, it dropped to 68 Shore A and was 65 Shore A after the second week. Except for percent elongation, the slight drop in physical properties at 392°F was similar to that experienced at 302°F after one week. These values were 665 psi tensile strength, 44 pli tear strength, and 30% ultimate elongation. They remained almost unchanged after two weeks at 392°F and were determined to be 665 psi tensile strength, 47 pli tear strength, and 43% ultimate elongation.

At 437°F testing, the reduction in percent elongation was more noticeable, with physical properties being 550 psi tensile strength, 56 pli tear strength, and 27% ultimate elongation after exposure for one week. Hardness decreased slightly to 68 Shore A. Percent elongation continued to decline after two weeks' exposure. At this time the values were 607 psi tensile strength, 38 pli tear strength, and 16% ultimate elongation. Hardness again was 68 Shore A.

The steadily decreasing percent elongation at this point was thought to be due to the loss of silicone oil from the specimens, as tensile strength, tear strength, and hardness remained fairly constant. After six days of testing, there was some evidence of plasticizer migration on all the test specimens.

However, since the heat stability of this material was satisfactory, it was decided to investigate this product further as a potential solar collector sealant candidate.

Experimental Formulation 7032-009

This material was based on Kaneka MS polymer, a polyoxypropylene glycol with terminal silane functionality, which was supplied by Kanegafuchi Chemical Company of Osaka, Japan. It was cured with a combination of zinc oxide and dibutyltin dilaurate.

The polymer was compounded with ground calcium carbonate while the accelerator was a paste of a phthalate ester vehicle and zinc oxide. The pigment volume concentration was made as high as possible (35%) to provide reinforcement. Many levels of zinc oxide in combination with dibutyltin dilaurate were evaluated before a level was found which gave the best physical values. Since this product cures via a silane condensation mechanism, it also was cast against silicone release paper inside a metal mold. The sheet, which was 80 mils thick, was removed from the metal mold after 16 hours at ambient temperature and allowed to cure in contact with the silicone paper for an additional 16 hours. It was then subjected to a heat cure of 120°F for 24 hours and 180°F for 16 hours. Tensile and tear specimens were then cut according to ASTM D412. At this time the material was resilient and

elastomeric. Physical properties were 120 psi tensile strength, 27 pli tear strength, 43% ultimate elongation, and 36 Shore A hardness.

Specimens were placed in the ovens at all three test temperatures. All degraded severely after 20 hours, even at 302°F. At this time the material had changed from a neutral tan color to a dark brown and surface cracks had developed on most of the surface area. This darkening penetrated the samples for approximately 20 mils, while the inside of the samples had become a semi-liquid gelatinous mass. Results at the higher test temperatures were the same but more severe. Because of the severe thermal degradation of this material, the zinc oxide cure mechanism was abandoned in favor of a silane cure as reported in the following section.

Experimental Formulation 7032-015

The Kaneka MS polymer composition of 7032-015 was cured with aminosilane using dibutyltin dilaurate as a catalyst. Six different aminosilane levels were evaluated to determine the optimum cure ratio. The minimum level at which the polymer cured satisfactorily was chosen.

The polymer was mixed, degassed, and cast exactly the same as 7032-009. Initial physical properties were 230 psi tensile strength, 55 pli tear strength, and 270% ultimate elongation. These higher physical properties were also reflected in the higher hardness value of 52 Shore A as compared to 36 for 7032-009.

After four hours at the lowest test temperature, 302°F, the tensile specimens remained rubbery but had turned from white to yellow. Some specimens formed a tough, leathery, degraded skin with bubbles at the surface. Those at higher test temperatures exhibited similar behavior. After

six hours of heat exposure, all specimens had turned dry and brittle. No further work was done with Kaneka MS polymer as its potential seemed well below the standards required for thermal stability.

Experimental Formulation 7032-001

This composition was based on a polysulfide polymer manufactured by Products Research & Chemical Corporation. It was formulated in much the same way as 7032-009; that is, the proper amount of calcium carbonate was added to obtain the highest possible pigment volume concentration while still maintaining a pourable consistency to facilitate degassing. This was approximately 30% by volume. The minimum amount of manganese dioxide necessary to effect a complete cure was determined experimentally. A trace of tertiary amine was added as a catalyst.

In casting sheets, the material degassed readily and cured well in a closed, release-sprayed mold. After casting, the cured sheet was removed from the mold and allowed to further cure for 16 hours at ambient temperature. It then received a post cure of 16 hours at 120°F and 16 hours at 180°F.

Specimens were made in the usual manner and tested according to ASTM D412. Initial physical properties were as follows: 185 psi tensile strength, 36 pli tear strength, 110% ultimate elongation and 40 Shore A hardness. A set of specimens was then placed in each of the three dry heat ovens for testing at 302°F, 392°F, and 437°F.

After two hours at all temperatures, the specimens had lost elastomeric properties. For this reason no further work was done with similar compositions.

Experimental Formulation 7032-056

Ethylene propylene diene monomer (EPDM) rubber is known for thermal stability and resistance to UV degradation. In addition, it can be highly extended by processing type oils. It was therefore chosen for evaluation in this program. The formulated product was made in steps, using an EPDM/oil master batch in both base and accelerator components. EPDM was first extended with oil using a heated Baker-Perkins sigma blade type laboratory mixer. Calcium carbonate fillers, titanium dioxide, carbon black and solvent were then added.

The base component was then made by adding adhesion promoters, an amine accelerator, and a crosslinking agent. The accelerator was made by adding more solvent and an oxidizing agent dispersed in naphthenic oil to the above master batch. Considerable work was then done to optimize the amounts of crosslinking agent, amine accelerator, and oxidizing agent to achieve maximum physical properties. The minimum level of these ingredients that effected a good cure was chosen for use. Solvent was added to the formula to lower viscosity, and a tensile sheet was cast between sheets of silicone paper inside a metal mold. This produced a satisfactory casting, which was then air dried for 16 hours at R.T. After removal of the release paper, the material was force dried for 16 hours at 120°F, followed by post curing for 16 hours at 180°F.

One set of tensile specimens was then made for determination of initial physical properties and three sets of tensile specimens were made for high temperature exposure testing.

Initial physical properties were 259 psi tensile strength, 30 pli tear strength, 170% ultimate elongation, and 41 Shore A hardness.

After one week at 302°F, physical properties were 134 psi tensile strength, 24 pli tear strength, 77% ultimate elongation, and 41 Shore A hardness. After the first few days at 302°F, oil began migrating out of the specimens and they gradually decreased in elongation. This became more severe until the specimens had lost considerable flexibility after one week. The samples at 392°F and 437°F exhibited the same phenomena except that oil loss was more severe. These samples cracked and became dry after exposure for one week and were unsuitable for testing. Continued exposure at 302°F degraded the samples to such a degree that they were not suitable for testing after the second week. However, it was felt that by selection of a less volatile plasticizer as well as careful selection of fillers and curatives, a relatively inexpensive sealant might be developed using this polymer system for somewhat lower temperature and performance applications. This material was therefore chosen for further reformulation and evaluation in subsequent work.

Experimental Formulation 7032-060

This sealant candidate was based on an aliphatic amine-cured epoxy. Flexibility was achieved by using high molecular weight amine-terminated polyoxypropylene curing agents which were obtained from the Jefferson Chemical Company. Initial formulation work with this system began with the curing of a medium molecular weight polyoxypropylene diepoxide with low molecular weight diamines such as hexamethylene diamine or 4,4'-diaminodiphenylmethane (MDA). However, the toxicity and difficult handling properties (many aromatic amine curing agents are solids) of these amines made formulation and use of this material difficult.

Formulations were made with high molecular weight aliphatic diamines which were cured with a typical 200 molecular weight diepoxide based on Bisphenol A. The particular diamine chosen had a molecular weight of approximately 2000 and was difunctional. Precipitated calcium carbonate was chosen as a filler, which gave a slight reinforcing effect to the cured material.

Several tertiary amine catalysts were evaluated and 2,4,6-dimethylmethyl-amino phenol was chosen as the most effective. Amine/epoxide ratio, filler content, and catalyst concentration and type were optimized to obtain the strongest and most elastomeric polymer. At this time, worklife and handling properties of the product were satisfactory.

After mixing and degassing this material at 120°F and curing it for 16 hours at R.T., the cast 80 mil sheet was removed from the mold and post cured for 16 hours at 120°F, followed by 16 hours at 180°F.

Tensile specimens were then cut and tested according to ASTM D412. Initial physical properties were 258 psi tensile strength, 42 pli tear strength, 160% ultimate elongation and 50 Shore A hardness. Samples were placed at all three test temperatures. However, after only two hours at 392°F and 437°F, samples had changed in color from the original milky white to medium brown. After two hours at 302°F, this material appeared unchanged in properties, but had also turned slightly brown. After 24 hours at 302°F, the surfaces of the samples had become tacky. The 392 and 437°F samples had turned dry and brittle. At 76 hours at 392 and 437°F, the samples had become more brittle with the interior of the material full of cavities which contained a dark brown liquid. Upon continued exposure at 302°F, the samples became very soft and sticky. They were dark brown in color and had elongated

noticeably. None of these specimens were tested for physical properties, because of the obvious rapid thermal degradation.

No further work with epoxy-based sealants was performed.

Experimental Formulation 7032-067

Since epichlorohydrin rubber is presently used as a preformed gasket in various solar collector applications, this elastomer was a logical choice for formulation into a potential collector sealant. At the time when the proposal for this contract was written, a low molecular weight, liquid epichlorohydrin polymer was available from Hercules. However, by the time work on the contract actually began, the material had been withdrawn from the market. Thus, formulation of an epichlorohydrin sealant based on currently available materials involved extension of a high viscosity epichlorohydrin rubber with solvent. This was necessary to reduce viscosity to a manageable level. A low solids coating was formulated first for evaluation purposes.

Epichlorohydrin propylene oxide copolymer was dissolved in a 50/50 blend of xylene and tetrahydrofuran. Heat stabilizer and carbon black were then ball milled into solution. The curing agent for this base, anhydrous piperazine, was dissolved in anhydrous ethanol to make a suitable curing agent. Molecular sieve 3A was added to this accelerator to insure dryness.

Various ratios of curing agent were evaluated until the best overall physical properties were obtained.

The resulting formula was 16% solids by weight. A tensile sheet was made by coating multiple layers onto silicone release paper. The coating was allowed to cure overnight at ambient temperature between each coat. A slight degassing of the mixed material was needed to remove air trapped during

mixing. Approximately a dozen coats were needed to produce a film of the required 80 mil thickness.

Initial physical properties were 602 psi tensile strength, 157 pli tear strength, 260% ultimate elongation, and 52 Shore A hardness.

A set of test specimens was made for each of the three test temperatures. After three hours at 302°F, the tensile specimens had flowed and elongated. Surface cracks were also present. After another three hours, they had degraded and flowed to such an extent that they were not testable.

As water can interfere with the curing mechanism of this material, water analysis of the base and accelerator was performed. The accelerator was found to have almost one percent water, despite the molecular sieve that had been added. A new accelerator was made which had less than 0.1% water.

Using the multiple layer technique again, another tensile sheet was cast, but only of 20 mil thickness. This sheet was cured in the same manner as previously. After three hours at 302°F, this formula exhibited the same phenomenon.

Several more specimens were made using various levels of piperazine and several fillers as stabilizers, but the results were almost unchanged. Because of poor thermal stability, no further work was done with epichlorohydrin rubber.

PR-1710

The excellent high temperature performance of Viton® fluoroelastomer is well known. For this reason, a Viton®-based coating, PR-1710, manufactured by Products Research & Chemical Corporation, was chosen for evaluation.

PR-1710 is a low-solids product, and a cast tensile sheet of

approximately 80 mils thickness was made in a similar manner to the epichlorohydrin sheet using a multiple coat technique. The material was allowed to cure for 16 hours at ambient temperature between each coat. It was then separated from the silicone release paper and cured 16 hours at 120°F, followed by 16 hours at 180°F.

Test specimens were cut from this 80 mil slab according to ASTM D412 and were tested at the three test temperatures.

Initial tensile properties for this material were 1052 psi tensile strength, 112 pli tear strength, 280% ultimate elongation, and 63 Shore A hardness. After one week at all test temperatures, hardness and elongation for all specimens remained relatively constant, but tensile strength increased dramatically, indicating that the PR-1710 was continuing to cure at these elevated temperatures.

After exposure at 302°F for one week, results were 1764 psi tensile strength, 140 pli tear strength, 160% ultimate elongation, and 80 Shore A hardness. This represented almost a 70% rise in tensile strength.

With 392°F exposure, results were 1978 psi tensile strength, 152 pli tear strength, 150% ultimate elongation, and 80 Shore A hardness. This was a 90% tensile strength rise, and was almost the ultimate strength that this material obtained in all three temperature environments.

Physical properties after 437°F exposure were somewhat less than those exposed to 392°F. Results were 1660 psi tensile strength, 152 pli tear strength, 113% ultimate elongation, and 80 Shore A hardness.

After two weeks in test, tensile strength for specimens at 302°F increased, but remained approximately the same after exposure at 392°F and 437°F. Results for 302°F exposure were 2158 psi tensile strength, 145 pli

tear strength, 150% ultimate elongation, and 76 Shore A hardness. Results for 392°F exposure were 1980 psi tensile strength, 183 pli tear strength, 133% ultimate elongation, and 76 Shore A hardness.

Since this material showed little deterioration after two weeks' exposure, these tests were continued for an additional week. At this time, all tensile values fell off slightly. After three weeks at 302°F, results were 1655 psi tensile strength, 135 pli tear strength, 123% ultimate elongation, and 80 Shore A hardness. After three weeks at 392°F, tensile strength was 1803, tear strength was 143 pli, ultimate elongation was 120%, and Shore A hardness was still 80. The three week 437°F exposure test results were 1675 psi tensile strength, 151 pli tear strength, 120% ultimate elongation, and hardness remained 80 Shore A.

The largest average drop in tensile strength after exposure was 20%, while tear strength, ultimate elongation and hardness remained almost unchanged.

Visual observations of PR-1710 during this testing were made at regular intervals. No noticeable changes took place in the samples. The specimen surface remained smooth and shiny with no evidence of color change or degradation. Based on excellent resistance to high temperature degradation, this material was selected for further evaluation.

Experimental Formulation 7032-003

This sealant was compounded from a commercially available thiol-terminated polyoxypropylene-based polymer (PM Polymer, Phillips Petroleum Co.). After establishing the desired degree of loading with precipitated calcium carbonate to promote reinforcement, the compounded base was cured with

a dispersion of manganese dioxide in a phthalate ester plasticizer. After degassing, a cast tensile sheet of 80 mils thickness was made using a closed metal mold. It was then allowed to cure for 16 hours at room temperature after removal from the mold. The cured sheet was post cured for 16 hours at 120°F followed by 16 hours at 180°F.

Tensile specimens were cut according to ASTM D412. Initial values were 99 psi tensile strength, 33 pli tear strength, 330% ultimate elongation, and 43 Shore A hardness.

After only two and one-half hours at all test temperatures, all specimens had turned brittle and dry on the outside, and the inside was a pink, dry powder. Because of the poor tolerance to high temperature, no further work was done with PM Polymer.

PR-810

This sealant, supplied by Products Research & Chemical Corporation, is based on an acrylate type rubber which has been specially formulated for stability at high temperatures. It is approximately 50% solids by weight, is thixotropic and as such was unsuitable for casting bubble-free tensile specimens. To reduce viscosity, the thixotropic agent was excluded from the formula. Approximately 25% of the reinforcing filler was also removed. Upon compounding this new formula, viscosity was still unacceptably high, and the addition of 25% more solvent produced a processable material.

Due to the high solvent content, this material was degassed for only a brief time. This removed almost all trapped air, and the remainder was removed when a tensile sheet was made. A metal mold was used for casting the tensile sheet, but the top was left open to permit bubble release and solvent evaporation. The material was applied to the mold in five successive coats

with a 16-hour dry time at room temperature between each coat. After the final coat, the tensile sheet was cured for 48 hours at ambient temperature followed by 16 hours at 120°F and 16 hours at 180°F. At this time, the cast sheet contained no residual solvent, and tensile and tear specimens were cut according to ASTM D412.

Initial physical properties were 580 psi tensile strength, 162 pli tear strength, 265% ultimate elongation, and 80 Shore A hardness. After one week at 302°F, the samples were almost unchanged. Tensile strength was 505 psi, tear strength was 155 pli, ultimate elongation was 240%, and hardness was 85 Shore A. Samples in the 392°F and 437°F ovens were examined daily. After three days at these temperatures, the specimens had lost most of their elasticity, but otherwise appeared unchanged. By six days, all specimens at these two higher temperatures were dry and brittle and could not be tested. The 302°F samples, after two weeks' exposure, had many minute cracks on the surface, had turned to a hard, almost brittle rubber, and had lost their surface gloss. These specimens were at best borderline in performance at 302°F and no further work was done with PR-810.

PR-812

PR-812 is based on an acrylate rubber similar to that used in PR-810. However, in PR-812 cure takes place via an amine type accelerator rather than solvent evaporation. Reformulation of this product was again necessary to obtain a low-viscosity product which could be degassed easily in order to produce a void-free tensile sheet. The thixotropic agent and a substantial portion of the reinforcing filler were deleted from the formulation. The level of solvent was doubled to produce a material which flowed fairly

easily. In casting sheets, the base was first degassed, then mixed with the accelerator, and then degassed again. A metal mold was used, but the cover was left off to facilitate solvent release. Five successive coats of material were flowed into the mold. A 16-hour ambient cure was allowed between each coat. After the application and ambient cure of the fifth coat, the cast sheet was removed from the mold and allowed to air dry for 24 hours at room temperature. It was then post cured for 16 hours at 120°F followed by 16 hours at 180°F. At this time the physical properties were determined to be 608 psi tensile strength, 174 pli tear strength, 283% ultimate elongation, and 84 Shore A hardness. After several days testing at high temperature, all test specimens became hard and brittle with surface crazing present. At six days, the specimens, even at the lower test temperature of 302°F, had cracked severely, precluding physical testing.

No further work was done with acrylate rubber as both the vulcanized and unvulcanized types exhibited inadequate thermal stability.

PR-1933

Original work proposed for this contract included the evaluation of PR-1933 as a potential solar collector sealant. As an aid to lowering viscosity for preparing sheets and to lower cost, the PR-1933 base was compounded with 30 parts of ground calcium carbonate to replace iron oxide. This provided a flow-type base for purposes of casting tensile specimens. In initial work with this modified base, application life was found to be short. Ten organometallic catalysts were evaluated in order to slow cure rate and achieve improved stability. However, a suitable replacement for the catalyst system in use was not found.

In the casting of test sheets for high temperature testing, the curing agent/catalyst mixture was combined with the modified PR-1933 base. After mixing completely, the material was degassed and poured between two sheets of silicone release paper in a metal mold. This procedure was used because this material does not cure adequately in a closed mold. The resulting 80 mil sheet was left in the mold for 24 hours at ambient temperature. At this time, a partial cure had occurred. The sheet was then allowed to cure for another 24 hours at ambient temperature before removing the release paper. It was then cured for 24 hours at 120°F, followed by 24 hours at 180°F.

After the 180°F post cure, physical properties were 114 psi tensile strength, 23 pli tear strength, 577% ultimate elongation, and 23 Shore A hardness.

After ten days at 392°F, physical properties were as follows: 200 psi tensile strength, 33 pli tear strength, 430% ultimate elongation, and 33 Shore A hardness. As in the case of the Viton®-based sealant, PR-1710, this material was also continuing to cure up to this point. Thus the significant reduction in ultimate elongation was believed to be the result of further curing rather than hardening produced by degradation. This was confirmed by physical property values after ten days at 437°F, which were 210 psi tensile strength, 30 pli tear strength, 445% ultimate elongation, and 20 Shore A hardness. These probably represent the ultimate physical properties of this formulation.

After three weeks at 392°F, tensile strength changed from 200 psi to 179 psi. Tear strength was 34 pli. Hardness increased by two points to 22 Shore A, and percent ultimate elongation decreased from 430 to 360%. After three weeks at 437°F, tensile strength of the PR-1933 modification above had fallen

from 210 psi to 189 psi, while hardness increased from 20 to 27.

Visual observation of the samples during testing confirmed that no gross physical changes had taken place in the specimens. There was no evidence of blistering, fading, cracking or color change. However, in order to promote compatability between the curing agents and PR-1933 base, a small amount of silicone oil is present in the curing agent. Visual observation of the test samples established that this small amount (less than 0.5%) of oil was migrating out of the samples on heat exposure. However, the excellent retention of physical properties upon heat exposure made this material a candidate for further evaluation in subsequent contract phases.

General Electric 1200

This one-part material cures through release of acetic acid. An 80 mil tensile sheet was cast between sheets of silicone paper. After 24 hours at ambient temperature, the cast sheet was separated from the silicone paper and allowed to cure for one week at ambient temperature. It was then subjected to 120°F/50% RH for 24 hours followed by 180°F/50% RH for 24 hours. At the end of this time test specimens were made. Initial tensile strength and percent ultimate elongation were high as compared to most of the other silicones. Physical properties were 494 psi tensile strength, 72 pli tear strength, 555% ultimate elongation and 25 Shore A hardness. After one week at 392°F and at 437°F, a noticeable increase in hardness and decrease in percent ultimate elongation were noted with tensile strength and tear strength remaining relatively unchanged. These values were 528 psi tensile strength, 72 pli tear strength, 440% ultimate elongation, and a hardness of 37 Shore A at 392°F. At 437°F, physical properties were 489 psi tensile strength, 72 pli tear

strength, 450% ultimate elongation, and 40 Shore A hardness.

After two weeks of high temperature exposure, tensile strength fell slightly and percent ultimate elongation continued to decrease with tear strength and hardness changing only slightly, except at 437°F where tear strength fell to 63 pli. The values recorded were 427 psi tensile strength, 76 pli tear strength, 380% ultimate elongation, 38 Shore A hardness at 392°F, and 371 psi tensile strength, 63 pli tear strength, 380% ultimate elongation and 39 Shore A hardness at 437°F. No plasticizer migration, surface cracking, embrittlement or other degradation phenomena were noted in this material, as the physical specimens were unchanged in appearance from their original condition. This material was further evaluated for application and performance properties.

Dow Corning 732-CL-11

This one-part silicone had low initial percent ultimate elongation and tensile strength values. After heat aging, percent ultimate elongation decreased drastically as hardness increased, and the product became cheesy.

Initial physical properties were 138 psi tensile strength, 41 pli tear strength, 235% ultimate elongation, and 33 Shore A hardness. After one week at the test temperatures, physical values were 116 psi tensile strength, 33 pli tear strength, 230% ultimate elongation, and 32 Shore A hardness at 392°F, and 107 psi tensile strength, 32 pli tear strength, 210% ultimate elongation, and 31 Shore A hardness at 437°F. After two weeks at 392°F, results were 91 psi tensile strength, 29 pli tear strength, 190% ultimate elongation, and 30 Shore A hardness. At 437°F, values recorded were 113 psi tensile strength, 36 pli tear strength, 70% ultimate elongation, and 53 Shore A hardness. As

evidenced by the last set of physical properties where percent ultimate elongation dropped significantly while hardness increased, noticeable degradation had taken place at 437°F. Thus, this product was fair to poor in performance in Phase I testing. For comparison purposes with other silicone-based materials, this product was tested for application and adhesion properties.

Dow Corning 795

This one-part silicone performed well after one week at the test temperature of 392°F. After two weeks, however, severe degradation was noted and physical properties dropped. This was more pronounced at 437°F exposure.

Initial physical properties were 87 psi tensile strength, 33 pli tear strength, 270% ultimate elongation, and 31 Shore A hardness.

Results after one week heat aging were 69 psi tensile strength, 29 pli tear strength, 260% ultimate elongation, and 35 Shore A hardness at 392°F, and 73 psi tensile strength, 33 pli tear strength, 40% ultimate elongation, and 40 Shore A hardness at 437°F. After two weeks at 392°F, results were 70 psi tensile strength, 23 pli tear strength, 110% ultimate elongation, and 45 Shore A hardness. After two weeks at 437°F, this product had become dry and powdery and samples crumbled easily when handled. Physical properties could not be obtained for these specimens.

Adhesion, fogging and other properties of this product were investigated in Phase II.

Dow Corning X-3-7058

This was an experimental one-part silicone-based product obtained from

Dow Corning. At 392°F, physical integrity of the test specimens was maintained for a short time, but after two weeks at both 392°F and 437°F, the material had doubled in hardness while decreasing severely in percent ultimate elongation. Performance was similar to that of Dow Corning 732-CL-11.

Initial physical properties were 160 psi tensile strength, 42 pli tear strength, 240% ultimate elongation, and 32 Shore A hardness.

Results at two weeks were 132 psi tensile strength, 35 pli tear strength, 120% ultimate elongation, and 44 Shore A hardness at 392°F, and 128 psi tensile strength, 37 pli tear strength, 60% ultimate elongation, and 54 Shore A hardness at 437°F.

After three weeks, these values were 143 psi tensile strength, 32 pli tear strength, 130% ultimate elongation, and 44 Shore A hardness at 392°F and 167 psi tensile strength, 46 pli tear strength, 50% ultimate elongation, and 70 Shore A hardness at 437°F.

No surface cracking, plasticizer migration, or other phenomenon were noted during testing. Evaluation of this product was also continued in Phase II.

General Electric Silpruf

Initial physical properties of this one-part silicone were 88 psi tensile strength, 29 pli tear strength, 400% ultimate elongation, and 23 Shore A hardness. After several days of heat exposure at 392°F and 437°F, the product had become soft and was extremely tacky on the surface. Attempts to obtain any meaningful physical property values from these specimens were unsuccessful. Adhesion and other characteristics of this product were, however, evaluated in subsequent contract work.

Dow Corning 90-006-02

This is a two-part silicone-based product. After three weeks at 437°F, tensile strength and tear strength had fallen significantly, but hardness and percent ultimate elongation remained almost unchanged. In addition, no visible effects of heat degradation were observed in the test specimens.

Initial physical properties were 397 psi tensile strength, 40 pli tear strength, 145% ultimate elongation, and 47 Shore A hardness. After three weeks at 437°F, these values were 247 psi tensile strength, 15 pli tear strength, 128% ultimate elongation and 43 Shore A hardness.

This product was further tested for adhesion, fogging and other important properties in Phase II.

Dow Corning 3120

This material is also a two-part silicone-based product. Initial physical properties were 594 psi tensile strength, 55 pli tear strength, 110% ultimate elongation, and 60 Shore A hardness. After three weeks at 437°F, results were 495 psi tensile strength, 46 pli tear strength, 98% ultimate elongation and 63 Shore A hardness. This represented less than a 20% drop in tensile and tear strength with percent ultimate elongation and hardness, within experimental error, unchanged. No visible evidence of degradation was recorded for these test specimens and this product was further investigated in Phase II.

PHASE II

SECONDARY SCREENING AND REFORMULATION OF SELECTED POLYMERS

Under Sections 4.3, 4.4, and 4.5 of the contract, eleven sealants selected on the basis of performance in high temperature screening tests were further evaluated.

One of the most important determinations in this phase was fogging potential. This property was tested according to the AAMA specification. A sheet of the candidate material (30 to 40 mils x 6" x 12") was cast on aluminum foil and cured, preferably at room temperature. It was then pressed on the bottom surface and sides of a stainless steel vessel which was 4" in diameter by 6" high. The specimen was arranged so that the sealant area was entirely exposed on one side. A glass plate with a cold water condenser was placed on top of the container, which was then three-quarters immersed in a constant temperature oil bath at the desired test temperature. The specimen was heated in this manner for 16 hours with cold water (60°F) circulating through the condenser. At the end of this time, the glass plate was visually inspected for any signs of residue or "fog".

Adhesion in dry heat after exposure to 437°F was also evaluated on the three test substrates: glass, anodized aluminum, and galvanized steel. After applying 20 to 30 mils to the test panel, the materials were air dried for 24 hours and post-cured for 16 hours at 180°F. These samples were then subjected to 437°F for up to two weeks. These samples were removed from the oven daily and cooled to ambient temperature for visual examination and adhesion testing.

In a solar collector unit, many possible combinations of temperature, UV exposure, heat exposure and humidity exposure are possible. In order to simulate at least a good number of the possible environmental conditions,

adhesion specimens of each candidate sealant were made on glass, anodized aluminum, and galvanized steel for testing under different conditions of heat, humidity and UV exposure.

PR-1710

Though PR-1710 had superior high temperature stability, the problems associated with formulating this material into a suitable high temperature sealant proved to be formidable.

In order to reduce viscosity to a manageable level, the need for a stable, Viton®-compatible plasticizer was evident. After removing a substantial portion of the solvent, several plasticizers were evaluated in the formula as a means of reducing polymer viscosity. By this time, solvent content had been reduced to 30% by weight, which gave a workable consistency, but was still far too much for this type of a sealant. At this concentration, shrinkage from solvent loss would place excessive stress on the collector lites and/or sealant/substrate interface.

The three plasticizers selected for evaluation were PRC Permapol® silicone, a high molecular weight polycyanosiloxane polymer; Dow Corning FS-1265, a fluorosilicone polymer; and ditridecyl phthalate.

In actual formulation, each plasticizer was added to the PR-1710 base and mixed to uniformity. No immediate separation was noted in any of the formulas in amounts of up to 30 parts plasticizer per 100 parts Viton®.

All three plasticized bases were catalyzed, mixed, and degassed briefly before making flow-out sheets on silicone release paper. After curing at room temperature, these samples were peeled from the silicone paper, allowed to air dry, and then post-cured for 16 hours at 180°F. All specimens exhibited a

tackiness which was not present in the unplasticized material. Further, high temperature testing at 302°F proved that all of these plasticizers were incompatible with PR-1710 Part B, at least at the 30% by weight level based upon Viton® weight. Upon heat exposure, droplets of plasticizer formed on the surface of the specimens within 8 hours. By 16 hours, this migration was severe.

All three plasticizers were then added to the PR-1710 Part B at amounts of 5, 10, 15, 20, and 25% by weight based on the Viton® elastomer. Sheet specimens were again made and cured as described above.

After eight hours at 302°F, 392°F, and 437°F, when the sheets were examined for bleeding, all but the lowest (5%) level of ditridecyl phthalate showed severe plasticizer migration. In addition, a high weight loss was noted.

After eight hours at all test temperatures, the formulas with cyanosiloxane polymer had also migrated at levels above 15%. Below 15%, the Viton® remained soft and elastomeric with no evidence of bleeding.

The fluorosilicone-plasticized PR-1710 Part B exhibited the same phenomena as the cyanosiloxane plasticizer; that is, only below 15%, based on the weight of Viton® in the formula, did the plasticizer appear to be compatible.

Further, in retesting all samples at 437°F for one week, in almost all instances, surface cracking of the samples occurred, some bleeding was noted, and significant weight losses were recorded.

Adhesion results were as follows: adhesion to glass was fair, except after UV or water exposure. Adhesion to anodized aluminum and galvanized steel was also fair.

Fogging tests of all formulas were run concurrently with high temperature tests. The conclusions were that, even at incompatible (greater than 15%) levels of fluorosilicone and cyanosilicone plasticizers, these materials did not fog at 230°F. The results of silicone oil modified PR-1710 showed slight fogging even though these oils are rated as being stable up to 500°F. The more volatile plasticizers such as ditiidecyl phthalate and chlorinated paraffins exhibited severe fogging.

High temperature adhesion testing of these PR-1710 formulas produced fair to poor results. Adhesion was satisfactory on anodized aluminum, and changed little on this substrate after one week at all test temperatures. Adhesion to glass and galvanized steel was fair initially, but decreased to poor after one week.

Since PR-1710 could not be successfully modified to produce suitable handling properties and an acceptable cost, work with this material was discontinued.

Experimental Formulation 7032-056

In this phase of the contract, formula 7032-056, the sealant based on EDPM rubber, was reformulated. The amount of paraquinone dioxime (crosslinker) in the initial formula was much greater than the theoretical amount. Six different concentrations of crosslinker were evaluated while maintaining a constant amount of oxidizing agent. Small circular (2"-diameter by 40 mils) cast sheets were then prepared and cured at 180°F. Die cut dumbbell physical property specimens were then cut according to ASTM D412 and were heat aged in the oven at 302°F and examined for physical changes. The net results of this testing were that the minimum amount of curative

needed to produce a tough, elastomeric sealant was approximately 25% of the initial amount. Below this concentration the cured rubber became excessively tacky and thermoplastic. Above this quantity, brown stains of unreacted paraquinone dioxime appeared.

At this time, small 2"-diameter x 40 mil samples were again prepared for high temperature testing using the reduced paraquinone dioxime formula. These cast sheets were made and cured at ambient temperature for 24 hours to allow solvent escape. They then received a post-cure of 16 hours at 120°F followed by 16 hours at 180°F.

After one week at 302°F, this formula with reduced paraquinone dioxime, reduced oxidizing agent (lead dioxide), and an antioxidant exhibited almost the same behavior as previous formulas. Surface cracking was evident and the specimens were no longer elastic.

Next, several ingredients thought to contribute to heat instability were deleted from the formula. The first such ingredient to be deleted was an iron oxide filler. This ingredient was in the formula to promote adhesion. However, its effect on polymer stability proved to be negative. Reformulation of the base was accomplished leaving this ingredient out, and 2"-diameter x 40 mil cast sheets were made of this formula.

High temperature testing of these samples showed no significant improvement in performance; cracking, drying out, and embrittlement had only very slightly improved.

Next the amine accelerator, Diak #2, was deleted from the formula. Cast sheets were made and tested. The stability in high temperature was improved, but not significantly.

No additional work was carried out with this material because it was felt

that more rapid progress could be obtained in the area of silicone-based materials.

PR-1977

In high temperature testing, PR-1977 was satisfactory, but some silicone oil migration was evident in the specimens. However, even with the presence of silicone oil which is volatile at 437°F, fog tests were satisfactory; no fogging was evidenced at 230°F.

Adhesion test panels were made on glass, aluminum and galvanized steel. Initial adhesion was excellent on glass and poor on anodized aluminum and galvanized steel. After one week of heat, water/UV exposures, adhesion to glass under all conditions was excellent, while adhesion to galvanized steel was poor. Adhesion to anodized aluminum, while showing promise after four days in the water/UV and no water/UV conditions, failed after one week exposure.

To improve adhesion, six different silane additives were evaluated. The first one evaluated was gamma aminopropyltriethoxy silane. This was used at 0.5% and 0.25% by weight of the PR-1977. This silane reduced worklife from about one hour to less than 20 minutes. Catalyst level was therefore reduced to lengthen the worklife to about 50 minutes. Adhesion panels were prepared using these formulas, and results were the same for both concentrations of aminosilane; adhesion to glass after one week test was excellent under all conditions, while adhesion to anodized aluminum and galvanized steel was poor. These samples were kept in the ultraviolet test chamber for one month, but adhesion did not improve, as it sometimes does when silane adhesion promoters are used. In addition, a chalking was noted on these samples which

was not present in the unmodified material.

Various other combinations of epoxy, mercapto, phenyl, and vinyl silanes with aminosilane, were evaluated at a total silane level of 1.0%. The results were generally the same with all silane combinations; adhesion to glass remained excellent and adhesion to anodized aluminum and galvanized steel was poor. However, even though these formulas did not adhere well to anodized aluminum initially, after one week at 160°F on all panels the material generally exhibited fair adhesion. At least a dozen additives were evaluated to promote hydrolysis of the silanes and thereby improve adhesion. Small amounts of calcium carbonate, zinc oxide, talc, titanium dioxide, barium sulfate, and other fillers known to affect silanes were added. No benefit was achieved through use of these additives.

Overall adhesion then, was good to glass, fair to poor on anodized aluminum, and poor on galvanized steel. Further work with PR-1977 was not pursued because other silicone-based materials offered better physical properties and potential adhesion.

PR-1939

Another potential solar collector sealant candidate evaluated was PR-1939, a proprietary sealant based on a silicone polymer and manufactured by Products Research & Chemical Corporation.

In the immediate past, this product had not been produced due to unavailability of certain key raw materials. During the final stages of Phase II, alternative sources of supply for these materials became available. PR-1939 is therefore included in this portion of the report. An 80 mil sheet was cast in the same manner as PR-1933. After removal from the silicone

release paper, the specimen was cured for two days at ambient temperature followed by 16 hours at 120°F and 16 hours at 180°F.

Tensile and tear strength specimens were then cut from this sheet according to ASTM D412. One set of specimens was tested for physical properties before heat exposure and yielded the following values: 200 psi tensile strength, 41 pli tear strength, 600% ultimate elongation and 24 Shore A hardness.

The remaining tensile strength and tear strength specimens were subjected to high temperature testing.

After three weeks' exposure to 392°F, physical properties were 220 psi tensile strength, 38 pli tear strength, 580% ultimate elongation and 24 Shore A hardness.

Physical properties after three weeks' exposure to 437°F were similar to those at 392°F. These were 230 psi tensile strength, 40 pli tear strength, 590% ultimate elongation, and 25 Shore A hardness.

Thus, PR-1939 exhibited satisfactory heat stability in this testing.

ADHESION TESTING

Ten commercially available silicone-based sealants were evaluated for adhesion. No primers were used in these evaluations, except with Dow Corning's 3120 and 90-006-02. These candidates were evaluated both with and without Dow Corning's #1200 primer.

Dow Corning 732-CL-11

Dow Corning 732-CL-11 had good initial adhesion to glass. After four, six and eight days in heat/humidity/UV, this material was still adhering

well. At twelve days, however, a slight adhesion drop was noted, but adhesion was still good. Most of the testing resulted in a cohesive rather than an adhesive specimen failure. Testing on glass was continued for one month. At the end of this time, adhesion was the same as that at twelve days and was still rated good.

Adhesion of this product to unprimed anodized aluminum was also rated excellent. After five days' exposure, a slight drop in physical properties was evidenced due to water sensitivity, but the mode of failure was still cohesive. The material remained unchanged for the duration of the 30-day test. This was encouraging, as many times sealants will fail when exposed to heat in the absence of UV and/or water. Adhesion in this instance was equal to the UV/water value.

Initial adhesion on galvanized steel, however, was poor. After five to eight days, a very low level cohesive failure mode was recorded, and after two weeks, slight improvement was observed in water exposure, with no improvement in the other conditions.

Dow Corning 795

The next product to be evaluated was Dow Corning 795. Initial and long term (one month) adhesion to glass were excellent, with a cohesive failure resulting in all cases. No drop in physical properties from water exposure was noted, nor was any change in initial adhesion observed in any of the six test conditions. Likewise, adhesion to anodized aluminum was excellent, both initially and after one month exposure to water/heat. Though excellent initially, adhesion to galvanized steel decreased after two days in water exposure. The dry portion of the test panel maintained good adhesion for a

few days longer, but ultimately fell to the same low level adhesive type failure. Overall adhesion of this material was satisfactory, but poor performance at high temperatures, contraindicates this product as a candidate for sealing solar collectors.

Silpruf

Adhesion of Silpruf® (General Electric one-part silicone) to glass, anodized aluminum and galvanized steel was excellent initially. After twelve days of UV/water exposure on glass, however, the material became soft and swollen even though it maintained adhesion. After one month, swelling had increased but the material still maintained good adhesion. In general, adhesion of this material to glass was poorer where exposure to UV was the least. Adhesion to anodized aluminum was still excellent after one month, whereas the sealant failed in adhesion to galvanized steel in a matter of a few days.

General Electric 1200

General Electric 1200 exhibited good adhesion to glass. Adhesion was excellent initially, and after ten days of water/UV testing, only a slight decrease in adhesion was noted. Though adhesion remained satisfactory under most other conditions, analysis showed many areas of slight adhesive failure, indicating a phenomenon that was greatly influenced by environmental conditions. After ten days of elapsed testing, adhesion began to increase until it was again at a satisfactory level after 33 days. This same phenomenon was not observed on the remaining two substrates. Adhesion to anodized aluminum was excellent and remained so for the entire test period.

Low level cohesive failure was observed initially on galvanized steel, and this also remained unchanged for the 30-day test period.

Dow Corning 90-006-02

Dow Corning 90-006-02 aerospace sealant was first evaluated without a primer. As expected from the literature, adhesion to all three test substrates was poor. With their recommended Primer #1200, adhesion was excellent to glass and anodized aluminum, but only fair to galvanized steel. After 30 days, adhesion to glass in all six environments was still excellent, and the same was true on anodized aluminum. After only two days of testing, however, adhesion to galvanized steel changed from excellent to a low-level, cohesive failure. In an effort to improve these results, a primer from Products Research & Chemical Corporation, PRC Primer #6, was also evaluated with this material. Results with this primer were somewhat erratic in terms of developing and/or losing adhesion. While adhesion to glass was good initially and in all conditions after one month testing, adhesion to anodized aluminum was poor initially, but increased to the point where it was good after two days. After 30 days, it was still good. Results on galvanized steel, however, were not improved; initial adhesion was good, but after three days, complete adhesive loss of adhesion was observed. Thus, no improvements were obtained with the use of this primer.

Dow Corning 3120

The second two-part sealant to be evaluated was Dow Corning's 3120. This material exhibited outstanding adhesion to all three substrates with the use of Dow Corning Primer #1200. Both initially, and after one month of test,

adhesion was excellent in all conditions of water/heat/UV. It was noted that this material had a slight tendency to chalk in water exposure.

PRC Primer #6 was also evaluated with this material. This combination proved to be unreliable, as adhesion loss and pick-up were observed; initial adhesion to glass was good. After two days, adhesion was poor with an adhesive separation to the substrate being recorded. After six days, adhesion picked up and was good at the end of the test period. Initial adhesion to galvanized steel was poor, but picked up after six days, and was good after thirty days. Similarly, complete adhesive failure to anodized aluminum was recorded before testing, but after two days, cohesive failure was noted, and adhesion was still good after one month. There was no advantage in the use of this primer.

Dow Corning 3120 appears to be a viable solar collector sealant when used with their #1200 primer.

PR-1939

At the end of Phase II testing, PR-1939 appeared to be a viable candidate for a solar collector sealant.

Adhesion to glass, galvanized steel, and anodized aluminum under water and UV conditions was evaluated in this phase. This PR-1939 formula exhibited satisfactory adhesion to all substrates under all conditions of water and UV exposure.

High temperature adhesion testing of this PR-1939 sealant formula was also evaluated. Adhesion panels were made on all three test substrates and exposed to dry heat of 302°F, 392°F and 437°F. Observations were made daily to examine the specimens for any adhesion loss or physical degradation. None

was noted, however, and at the end of three weeks at all temperatures, adhesion to all substrates was satisfactory.

A simulated solar collector was constructed using this product. The unit was made in a configuration similar to a double pane insulating glass unit. This particular collector was fabricated using 3/16" thick glass lites which measured 14" x 20". These were stacked one on top of the other with regular aluminum insulating glass spacers separating the glass around the periphery of the lites. A 14" x 20" sheet of galvanized steel was painted with flat black paint and placed under the bottom lite to absorb heat. Thermometers were positioned in between both lites to record temperatures during cycling. The entire unit was then held together with suitable clamps while all spacers were sealed with the candidate sealant.

A hole was drilled in each of the insulating glass spacers and a 12" x 1"-diameter metal drying tube with a small diameter nozzle was fitted in each spacer. These tubes were filled with 'Drierite' desiccant and further fitted with an aluminum membrane and a small cotton plug at each end. A small hole (10 to 20 mils) was made in each of these membranes to allow only a minimum amount of air movement. These tubes were then shielded from the sun and environment by a canopy of aluminum foil. This method proved to be quite adequate for testing, as no adverse effects from contamination were noted.

Though this particular material was somewhat short in worklife, construction of the unit was straightforward and relatively easy. The drying tubes were sealed in place with additional sealant.

Normal insulating glass units contain a dessicant to insure dryness of the interior glass surfaces. Due to the low relative humidity inside the unit, a pressure differential is created which creates a driving force for

moisture into the unit. If a desiccant were used in this simulated collector, water would continue to enter until the desiccant became saturated. During daylight hours when temperatures go as high as 200°F, water vapor would be driven back out. To prevent this needless migration of water and any associated contaminants, no dessicant was used inside the unit.

At the time of this report, this solar collector has successfully undergone five months of roof exposure. Generally, the unit has been running at temperatures approximately 100°F greater than ambient. For instance, on a day when the outside temperature was 102°F, the inside air space closest to the absorbing surface was 202°F while the outside air space was 173°F. On a particularly hot day with ambient temperature at 110°F, the inside air space was 222°F and the outside one was 190°F. A typical day might be 80°F with inside/outside lite temperatures running at 159/131°F.

PHASE III

MODIFICATION AND CONTINUED EVALUATION OF SEALANTS

Under this phase of the contract, fog testing was conducted according to the AAMA specification on all potential sealant candidates.

Dow Corning sealant 732-CL-11 fogged at temperatures greater than 200°F. Very small droplets of foreign material were observed to deposit on the glass collector plate.

Dow Corning 795 deposited a thin film of material at temperatures above 230°F, but could be considered satisfactory at temperatures below 230°F.

A thin film of material was also deposited by Silpruf at temperatures of from 200 - 210°F. This product may be satisfactory at lower temperatures, but in view of some water sensitivity that was observed in adhesion testing, this would need further examination.

General Electric 1200 deposited a thin, yellow film at temperatures above 200°F.

Fog test results for the two-component sealants were similar to those of the one-parts; at temperatures below about 210°F, they were all satisfactory.

In continuing evaluation of PR-1939, a base was made in which the polymer was degassed at 250°F for 12 hours prior to compounding with iron oxide. The net result of this improvement was the formulation of a material which was non-fogging at temperatures above 200°F.

Work was then continued with PR-1939. A study was undertaken to evaluate various metal driers in an effort to further improve heat stability and fogging resistance.

Stannous octoate, diphenylmercury dodecylsuccinate, lead naphthenate, lead octoate, cobalt octoate and naphthenate, phenyl mercuric acetate, a variety of

organometallic titanates, and other organometallic catalysts were evaluated for their reactivity. A great many of these materials had very little effect on the silicone reaction. In general, mercury, calcium, and zirconium driers inhibited the cure to such a degree that the mixed material was still liquid after 48 hours at ambient temperature. Iron and chromium also had an inhibiting effect on the cure rate. A proprietary tin catalyst was found (see Example, i.e., U. S. Patent 3,781,315 issued to Products Research & Chemical Corp.) which gave a good worklife (approximately one hour). This material produced a good elastomer in three to four hours.

Tensile sheets of this formula were made and cured at 180°F. High temperature testing confirmed that the physical properties were unchanged from the previous formula. Initial and water/heat/UV adhesion was repeated, and was still satisfactory. Fogging potential was tested and found to be 245°F, a significant improvement over the previous value of 200°F.

A simulated solar collector was made of this formula, and at the time of writing, was performing excellently after four months of roof exposure with temperatures ranging from 55°F to 204°F.

A summary of Phase III sealant performance is given in Table III.

PHASE IV

SEALANT FINALIZATION

Though performance properties were satisfactory, PR-1939 was somewhat difficult to mix because the base was a thixotropic paste consistency and the accelerator was a low viscosity liquid. Accelerator stability was also investigated to determine if improvement was possible.

Several hydroxyl bearing materials such as isopropyl alcohol, cellosolve, isobutanol, and low molecular weight polypropylene glycol were evaluated in the PR-1939 formula as a means of stabilizing the silane hydrolysis. Almost all of these materials worked to some extent, with the higher molecular weight hydroxyl bearing compounds inhibiting the cure rate to a greater degree. This was expected, as these additives must diffuse from the sealant before a cure can be effected. As a compromise between cure rate and stability, a low molecular weight alcohol was chosen for use. By adding this to the silane mixture, an accelerator with improved stability was produced. The worklife of this material was approximately one hour.

Again, high temperature and adhesion testing proved that the physical and adhesive properties of the sealant had not been changed.

Silicone oils had been proven to be volatile in earlier testing at 437°F, but this did not affect fogging temperature of earlier formulas. With this in mind, a thixotropic accelerator was formulated using silicone oil.

Precipitated calcium carbonate was used as a thixotropic agent with a moderate amount of silicone oil to act as a carrier vehicle. The product so formulated was moderately stable and was very easy to mix. High temperature testing even at 302°F did confirm, however, that the oil was evaporating at this temperature. While this apparently did not influence short term AAMA fog

testing, it is anticipated that long range effects of this oil could be detrimental to both fogging and adhesion.

Fumed silica was then evaluated in the base to increase thixotropy since the mixed material was not sufficiently thixotropic. As expected, this made the base more thixotropic, but it also was heavy and difficult to mix. This problem was solved by using silane treated fumed silica. The resulting material was thixotropic and not quite as heavy as the regular fumed silica material. In addition, this specially treated silica imparted a 20% reduction in moisture vapor transmission rate to the cured silicone. High temperature heat resistance, high temperature adhesion, and UV/water adhesion testing were unchanged.

Experiments were then conducted to determine if the amount of alcohol required for stability could be reduced, as it lengthened cure time and the amount had only been chosen arbitrarily in earlier work. The accelerator was approximately 15 to 20 percent alcohol by weight. Various levels were evaluated and it was determined that alcohol concentration could be reduced to 5 percent by weight. This reduction resulted in a significant decrease in cure through time in thick sections. Worklife and cure remained the same, about one hour, with the appropriate amount of organotin catalyst.

Thus, at the conclusion of the contract, two possible PR-1939 modifications existed: a product having a liquid accelerator and a material having a paste accelerator. The material with the liquid accelerator offers the best performance properties. Careful blending with the base is required to afford a complete mix. The modification having a thixotropic accelerator has improved mixing and application properties. While fogging potential may have been slightly impaired, the long term effects of this oil on performance have

not been established.

During this phase of the contract, testing of seven commercially available polysiloxane sealants was continued. Handling and application properties of these products were found to be satisfactory.

Low temperature flexibility of all seven sealants at -20°F was also found to be satisfactory.

All commercial sealants were tested for adhesion to anodized aluminum, glass and galvanized steel at 437°F. It was found that up to three weeks of continuous heat exposure, all materials possessed satisfactory adhesion to all substrates. After three weeks, however, adhesion loss was noted on some substrates. Thereafter, a steady decline in adhesion was recorded.

Overall results then, were unchanged from Phase III, where Dow Corning 90-006-02, and 3120 and General Electric 1200 exhibited good to superior performance in fogging, adhesion, low temperature flexibility and high temperature adhesion and heat exposure testing, and were considered viable candidates for a solar collector sealant along with PR-1939.

A summary of test results for all sealants evaluated in Phase IV is given in Table IV.

Formulations used to evaluate the properties of candidate elastomers are shown in Table V. Sources and descriptions of raw materials used in the formulations are given in Table VI.

SUMMARY

Ten elastomeric polymer types were formulated into sealants and examined for their retention of properties after heat exposure.

Ten commercial one-component and two-component sealants based upon polysiloxane rubbers were also evaluated for heat resistance, application properties, fogging potential and adhesion when applied to galvanized metal, aluminum and glass. The effects of exposure to water, air and ultraviolet light at 140°F on adhesion retention was determined.

The fluorinated elastomer, Viton® B, showed the highest retention of physical properties at elevated temperatures. Polysiloxane formulations were the next best, while EPDM rubber showed promise at lower temperatures. Both the fluorinated material and the ethylene propylene diene rubber required extensive dilution with solvent and plasticizers for easy handling--reducing their usefulness and performance.

Based upon relative retention of physical properties after heat aging, fogging resistance, and adhesion retention (with primers, if necessary), candidate solar collector sealants were Dow Corning 90-006-02 and 3120, General Electric 1200, and Products Research & Chemical PR-1939.

TABLE I

PHASE I: HIGH TEMPERATURE TESTING OF CANDIDATE MATERIALS

GENERAL SUMMARY

<u>Polymer Number</u>	<u>General Description</u>	<u>Retention of Physicals After</u> <u>High Temperature Exposure</u>		
		<u>302°F</u>	<u>392°F</u>	<u>437°F</u>
Sylgard 184	Silicone (control)	Pass	Marg.	Marg.
PR-1977	Silicone	Pass	Pass	Pass
7032-009	Silane-terminated polyoxypropylene	Fail	Fail	Fail
7032-015	Silane-terminated polyoxypropylene	Fail	Fail	Fail
7032-003	Thiol-terminated polyoxypropylene	Fail	Fail	Fail
7032-128	PRC polysulfide polymer	Fail	Fail	Fail
7032-056	EPDM Rubber	Marg.	Fail	Fail
7032-060	Polyoxypropylene-based epoxy	Fail	Fail	Fail
7032-067	Epichlorohydrin rubber	Fail	Fail	Fail
PR-812	Acrylonitrile rubber	Fail	Fail	Fail
PR-810	Acrylate rubber	Fail	Fail	Fail
PR-1710	Viton®	Pass	Pass	Pass
PR-1939	Silicone	Pass	Pass	Pass
Dow 795	Silicone	N/T*	Marg.	Fail
G.E. Silpruf	Silicone	N/T*	Fail	Fail
G.E. 1200	Silicone	N/T*	Pass	Pass
Dow 732-CL-11	Silicone	N/T*	Marg.	Fail
Dow 90-006	Silicone	N/T*	Pass	Pass
Dow 3120	Silicone	N/T*	Pass	Pass
Dow X-3-7058	Silicone	N/T*	Pass	Pass

*Not Tested.

TABLE II

PHASE I: HIGH TEMPERATURE TESTING OF CANDIDATE MATERIALS

SPECIFIC SUMMARY

Chemical Class.	Silicone	Silicone	Silane-terminated polyoxypropylene
Supplier	Dow	PRC	Kanegafuchi
Trade Name	Sylgard 184	PR-1977	Kaneka MSP-20S
<u>Physicals</u>			
<u>Initial:</u>			
Tensile	940	659	120
Tear	50	65	27
% Elong.	140	60	43
Hardness	45	73	36
<u>After 392°F:</u>	<u>2 Weeks</u>	<u>3 Weeks</u>	Failed after short time in high temperature testing; turned into dry, brittle material.
Tensile	189	579	
Tear	57	31	
% Elong.	33	93	
Hardness	57	62	
<u>After 437°F:</u>	<u>2 Weeks</u>	<u>3 Weeks</u>	
Tensile	199	516	
Tear	43	37	
% Elong.	50	73	
Hardness	50	63	
<u>Observations:</u>	Slight color change. Severe drop in tensile strength.	Slight color change. Silicone oil migration	Severe degradation at high temperatures

TABLE II

Chemical Class.	Silane-terminated polyoxypropylene	Thiol-terminated polyoxypropylene
Supplier Trade Name	Kanegafuchi Chemical Kaneka MSP-20S	Phillips Petroleum PM Polymer
<u>Physicals</u>		
<u>Initial:</u>		
Tensile	230	99
Tear	55	33
% Elong.	270	330
Hardness	52	43
<u>After 392°F:</u>		
Tensile	Failed after 4 hours	Failed after 5 hours
Tear	into a dry and brittle	into a form of dry and
% Elong.	material.	brittle material.
Hardness		
<u>After 437°F:</u>		
Tensile	Also failed in 4 hours.	Also failed in 5 hours.
Tear		
% Elong.		
Hardness		
<u>Observations:</u>		
	Severe degradation at high temperatures.	Severe degradation at high temperatures.

TABLE II

Chemical Class.	PRC polysulfide polymer	EPDM	Polyoxypropylene diamine/bis-epoxy
Supplier Trade Name	PRC Permapol polysulfide	B. F. Goodrich Epcar 346	Jefferson Chemical D2000 /Shell Epon 828

Physicals

Initial:

Tensile	185	259	258
Tear	36	29	42
% Elong.	110	170	160
Hardness	40	41	50

After 392°F:

Tensile	Failed after	Failed after	Failed after
Tear	3 hours.	7 days; also	26 hours. Dry,
% Elong.		sweated.	brittle material.
Hardness			

After 437°F:

Tensile	Also failed.	Also failed.	Also failed.
Tear			
% Elong.			
Hardness			

Observations:

Severe degradation at high temperatures.	Stiffening of material after 7 days.	Severe degradation at high temperature.
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TABLE II

Chemical Class.	Epichlorohydrin copolymer rubber	Acrylate rubber
Supplier Trade Name	Hercules Herchlor C	PRC PR-812
<u>Physicals</u>		
<u>Initial:</u>		
Tensile	602	608
Tear	157	179
% Elong.	260	283
Hardness	52	84
<u>After 392°F:</u>		
Tensile	Failed by melting into	Failed, becoming a dry,
Tear	an elongated shape with	brittle lump after
% Elong.	cracked surface after	5 hours.
Hardness	3 hours.	
<u>After 437°F:</u>		
Tensile	Failed as above.	Failed as above.
Tear		
% Elong.		
Hardness		
<u>Observations:</u>		
	Thermoplastic material. Some thermal degradation.	Severe degradation at high temperatures.

TABLE II

Chemical Class.	Acrylate rubber	Viton	Silicone	Silicone
Supplier	PRC	PRC	PRC	PRC
Trade Name	PR-810	PR-1710	PR-1933	PR-1939
<u>Physicals</u>				
<u>Initial:</u>				
Tensile		1052	114	200
Tear		112	23	41
% Elong.		280	577	600
Hardness		63	22	24
<u>After 392°F:</u>		<u>3 Weeks</u>	<u>3 Weeks</u>	<u>3 Weeks</u>
Tensile		1803	179	220
Tear		143	33	38
% Elong.		120	360	580
Hardness		80	22	24
<u>After 437°F:</u>		<u>3 Weeks</u>	<u>3 Weeks</u>	<u>3 Weeks</u>
Tensile		1675	189	230
Tear		151	34	40
% Elong.		120	300	590
Hardness		80	27	25
<u>Observations:</u>				
	After 6 days of high temperature testing, failed as a dry, brittle form.	Excellent appearance. No change.	No change in appearance. Slight sweating.	No change in appearance.

TABLE II

Chemical Class.	Silicone	Silicone	Silicone	Silicone
Supplier	G.E.	Dow	Dow	Dow
Trade Name	1200	732-CL-11	795	X-3-7058

Physicals

Initial:

Tensile	494	138	87	160
Tear	72	41	33	42
% Elong.	555	235	270	240
Hardness	25	33	31	32

<u>After 392°F:</u>	<u>2 Weeks</u>	<u>2 Weeks</u>	<u>2 Weeks</u>	<u>2 Weeks</u>
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Tensile	427	91	70	143
Tear	76	29	23	32
% Elong.	380	190	110	130
Hardness	38	30	45	44

<u>After 437°F:</u>	<u>2 Weeks</u>	<u>2 Weeks</u>	<u>2 Weeks</u>	<u>2 Weeks</u>
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Tensile	371	113		167
Tear	63	36		46
% Elong.	380	70		50
Hardness	39	53		70

Observations:

No visible evidence of physical degradation.	Became cheesy after testing.	Severe degradation. Samples crumbled. Dry and powdery.	Moderate loss of elastomeric properties. No visible changes.
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TABLE II

Chemical Class.	Silicone	Silicone	Silicone
Supplier			
Trade Name	GE Silpruf	Dow 90-006-02	Dow 3120
<u>Physicals</u>			
<u>Initial:</u>			
Tensile	88	397	594
Tear	29	40	55
% Elong.	400	145	110
Hardness	23	47	60
<u>After 392°F:</u>	<u>24 Hours</u>		
Tensile	Became soft	Not tested.	Not tested.
Tear	and tacky.		
% Elong.			
Hardness			
<u>After 437°F:</u>	<u>16 Hours</u>	<u>3 Weeks</u>	<u>3 Weeks</u>
Tensile		247	495
Tear		15	46
% Elong.		128	78
Hardness		43	63
<u>Observations:</u>	Possible reversion. Became soft and tacky.	No significant change in appearance.	No change in appearance.

TABLE III

SEALANTS TESTED UNDER PHASE II

Chemical Class.	Silicone	EPDM
Supplier	PRC	PRC
Trade Name	PR-1939	Development Product

Physicals

Initial:

Tensile	200	259
Tear	41	29
% Elong.	600	170
Hardness	24	41

<u>After 437°F:</u>	<u>3 Weeks</u>	(After 302°F)	<u>1 Week</u>
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Tensile	230	133
Tear	40	23
% Elong.	590	77
Hardness	25	41

Adhesion

Initial:

Glass	Good	Not tested
Galv. Steel	Good	Not tested
Anod. Alum.	Good	Not tested

After Heat/Humidity/UV:

Glass	Good	Not tested
Galv. Steel	Good	Not tested
Anod. Alum.	Good	Not tested

Low Temperature Flex	Pass	Not tested
MVT Rate	28 g./M ² /24 hours @ 70°F	1.5 g./M ² /24 hours @ 70°F
Fog Temp.	245°F	Not tested
Handling Properties:		
Worklife	1 hour	1 hour
Ease of Mixing	Good	Good
Ease of Application	Poor	Fair
Slump	Poor	Good

Observations:

No change in appearance.	Heat resistance marginal at moderate temperatures (302°F).
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TABLE III

Chemical Class.	Silicone	Viton®
Supplier	PRC	PRC
Trade Name	PR-1977	PR-1710
<u>Physicals</u>		
<u>Initial:</u>		
Tensile	659	1052
Tear	66	117
% Elong.	60	280
Hardness	73	63
<u>After 437°F:</u>	<u>3 Weeks</u>	<u>3 Weeks</u>
Tensile	579	1675
Tear	31	150
% Elong.	93	120
Hardness	62	80
<u>Adhesion</u>		
<u>Initial:</u>		
Glass	No adhesion	No adhesion
Galv. Steel	No adhesion	No adhesion
Anod. Alum.	No adhesion	No adhesion
<u>After Heat/Humidity/UV:</u>		
Glass	Good adhesion	No adhesion
Galv. Steel	Poor adhesion	No adhesion
Anod. Alum.	Poor adhesion	No adhesion
Low Temperature Flex		
MVT RATE		
Fog Temp.		
Handling Properties:		
Worklife	4 hours	
Ease of Mixing	Hard to mix, high viscosity	
Ease of Application	Fair	
Slump	OK	
<u>Observations:</u>		
	Adhesion poor. No change in appearance.	Excellent appearance. No visible changes.

TABLE IV

SEALANTS TESTED UNDER PHASES III AND IV

Chemical Class.	Silicone	Silicone
Supplier	PRC	PRC
Trade Name	PR-1939, Type I (fluid accelerator)	PR-1939, Type II (thixotropic accelerator)
<u>Physicals</u>		
<u>Initial:</u>		
Tensile	239	226
Tear	40	44
% Elong.	643	560
Hardness	32	27
<u>After 437°F:</u>	<u>3 Weeks</u>	<u>3 Weeks</u>
Tensile	248	224
Tear	42	52
% Elong.	710	433
Hardness	22	24
Fog Temp.	245°F	220°F
<u>Adhesion</u>		
<u>Initial:</u>		
Glass	Good	Good
Galv. Steel	Good	Good
Anod. Alum.	Good	Good
<u>After Heat/Humidity/UV:</u>		
Glass	Good	Good
Galv. Steel	Good	Good
Anod. Alum.	Good	Good
Low Temperature Flex		
MVT Rate	28 g./m ² , 24 hours @ 70°F	28 g./m ² , 24 hours @ 70°F
Fog. Temp.	245°F	220°F
Handling Properties:		
Worklife	1 hour	2 hours
Ease of Mixing	Poor	Good
Ease of Application	Fair	Good
Slump	Fair	Good
<u>Observations:</u>	Excellent. No visible change.	Excellent. No visible change.

TABLE IV

Chemical Class.	Silicone	Silicone
Supplier	Dow	Dow
Trade Name	90-006-02	3120
<u>Physicals:</u>		
<u>Initial</u>		
Tensile	397	594
Tear	40	55
% Elong.	145	110
Hardness	47	60
<u>After 437°F:</u>	<u>3 Weeks</u>	<u>3 Weeks</u>
Tensile	247	495
Tear	15	46
% Elong.	128	78
Hardness	43	63
<u>Adhesion</u>	(With DC Primer #1200)	(With DC Primer #1200)
<u>Initial:</u>		
Glass	Good	Good
Galv. Steel	Fair	Good
Anod. Alum.	Good	Good
<u>After Heat/Humidity/UV:</u>		
Glass	Good	Good
Galv. Steel	Fair	Good
Anod. Alum.	Good	Good
Low Temp. Flex	OK	OK
MVT Rate	Not tested	Not tested
Fog Temp.	200°F	Not tested
Handling Properties:		
Worklife		
Ease of Mixing	Good	Good
Ease of Appl.	Good	Good
Slump	Good	Good
<u>Observations:</u>	Slight chalking in adhesive testing (UV/ water).	No visible changes.

TABLE IV

Chemical Class.	Silicone	Silicone
Supplier	G.E.	Dow
Trade Name	1200	X-3-7058
<u>Physicals</u>		
<u>Initial:</u>		
Tensile	494	160
Tear	72	42
% Elong.	555	240
Hardness	25	32
<u>After 437°F:</u>	<u>2 Weeks</u>	<u>3 Weeks</u>
Tensile	371	167
Tear	63	46
% Elong.	380	50
Hardness	39	70
<u>Adhesion</u>		
<u>Initial:</u>		
Glass	Good	Not tested
Galv. Steel	Poor	Not tested
Anod. Aluminum	Good	Not tested
<u>After Heat/Humid./UV:</u>		
Glass	Erratic	Not tested
Galv. Steel	Poor	Not tested
Anod. Aluminum	Good	Not tested
Low Temperature Flex	OK	
MVT Rate	Not tested	
Fog Temp.	200°F	
<u>Handling Properties:</u>	(One-Part Material)	(One-Part Material)
Worklife	Not applicable	Not applicable
Ease of Mixing	Not applicable	Not applicable
Ease of Application	Good	Good
Slump	Good	Good
<u>Observations:</u>	No visible changes.	No visible changes.

TABLE IV

Chemical Class.	Silicone	Silicone	Silicone
Supplier	Dow	Dow	G.E.
Trade Name	732-CL-11	795	Silpruf
<u>Physicals</u>	See Table III	See Table III	See Table III
Initial:			
Tensile			
Tear			
% Elong.			
Hardness			
After 437°F:			
Tensile			
Tear			
% Elong.			
Hardness			
<u>Adhesion</u>			
Initial:			
Glass	Good	Good	Good
Galv. Steel	Poor	Good	Good
Anod. Aluminum	Good	Good	Good
After Heat/Humid./UV:			
Glass	Good	Good	Good
Galv. Steel	Fair	Poor	Poor
Anod. Aluminum	Good	Good	Good
Low Temperature Flex	OK	OK	OK
MVT Rate	Not tested	Not tested	Not tested
Fog Temp.	200°F	230°F	200°F
<u>Handling Properties:</u>			
Worklife	Not applicable	Not applicable	Not applicable
Ease of Mixing	Not applicable	Not applicable	Not applicable
Ease of Application	Good	Good	Good
Slump	Pass	Pass	Pass
<u>Observations:</u>	Loss of elastomeric properties.	No visible changes.	Very moisture sensitive. Softened and swelled.

TABLE V
SEALANT FORMULAS

Sylgard 184	
Proprietary polysiloxane base	100.00
Proprietary accelerator	10.00
PR-1977 (silicone polymer)	
Part B	
Silanol-terminated polymer (proprietary base)	100.00
Chopped glass	
Red iron oxide	
Part A	
Dibutyltin dilaurate catalyst	0.50
7032-009 (Silane-terminated polyether/zinc oxide cure)	
Part B	
Silane-terminated polymer (Kaneka MSP-20S)	100.00
Calcium carbonate	120.00
Part A	
Ditridecyl phthalate	4.25
Zinc oxide	6.75
Dibutyltin dilaurate	0.50
Part C	
Water	2.20
7032-015 (Silane-terminated polyoxypropylene/silane cure)	
Part B	
Silane-terminated polyoxypropylene (Kaneka MSP-20S)	100.00
Coated calcium carbonate	120.00
Part A	
Gamma aminopropyl triethoxy silane	11.00
Part C	
Dibutyltin dilaurate	0.50
7032-003 (Mercaptan-terminated polyoxypropylene/manganese dioxide cure)	
Part B	
Mercaptan-terminated polyoxypropylene (Phillips PM)	100.00
Calcium carbonate	200.00
Part A	
Diisodecyl phthalate	10.00
Manganese dioxide	20.00
Tetramethyl guanadine	Trace
7032-128 (Polysulfide, manganese dioxide cure)	
Part B	
Polymer (PRC Permapol® polysulfide)	100.00
Calcium carbonate	200.00
Part A	
Diisodecyl phthalate	10.00
Manganese dioxide	20.00
Tetramethyl guanidine	Trace

TABLE V

7032-056 (EPDM rubber/paraquinone dioxime and lead dioxide cure)

Part B	
Epcar 346 (EPDM)	100.00
Sunpar 2280 (oil)	460.00
Precipitated calcium carbonate	200.00
Coated, ground calcium carbonate	550.00
Titanium dioxide	5.00
Carbon black	20.00
Yellow iron oxide	30.00
Toluene	110.00
Diak #2	3.00
Paraquinone dioxime	13.00
Part A	
Epcar 346	11.00
Sunpar 2280	70.00
Precipitated calcium carbonate	22.00
Coated, ground calcium carbonate	60.00
Titanium dioxide	0.50
Carbon black	2.20
Yellow iron oxide	3.30
Toluene	25.50
Lead dioxide	44.00

7032-060 (Polyoxypropylene diamine/Bisphenol A epoxy)

Part B	
Bisphenol A epoxy (200 eq. wt.)	100.00
Part A	
Polyoxypropylene diamine (1000 eq. wt.)	420.00
Precipitated calcium carbonate	40.00
Ground calcium carbonate	380.00

7032-067 (Epichlorohydrin rubber/piperazine)

Part B	
Herchlor C (poly-epichlorohydrin)	100.00
Part A	
Piperazine	6.00

PR-1939 (Polysiloxane polymer base/proprietary accelerator)

Type I: Fluid	
Part B	
Proprietary polysiloxane base	129.00
Part A	
Proprietary accelerator	7.57
Ethanol	5.33

PR-1933

Part B	
Proprietary polysiloxane base	100.00
Part A	
Proprietary accelerator	10.00

TABLE V

PR-1939	
Type II (Thixotropic)	
Part B	
Polysiloxane base	129.00
Part A	
Accelerator	13.70
Dry ethanol	2.52
Precipitated calcium carbonate	16.12
General Electric 1200 Sealant	
One-part, polysiloxane-based sealant	
General Electric Silpruf	
One-part, polysiloxane-based sealant	
Dow Corning 795	
One-part, polysiloxane-based sealant	
Dow Corning X-3-7058	
Experimental, one-part, polysiloxane-based sealant	
Dow Corning 90-006-02	
Two-part, polysiloxane-based sealant	
Dow Corning 3120	
Two-part, polysiloxane-based sealant	
Dow Corning 732-CL-11	
One-part, polysiloxane-based sealant	
Dow Corning Primer #1200	
One-part primer used with Dow Corning 90-006-02 and Dow Corning 3120	

TABLE VI

RAW MATERIAL SUPPLIERS

<u>Material</u>	<u>Chemical Description</u>	<u>Supplier</u>
A-187	Glycidoxypopyl trimethoxysilane	Union Carbide Corp.
A-189	Mercaptopropyl trimethoxysilane	Union Carbide Corp.
A-1100	gamma-Aminopropyl triethoxysilane	Union Carbide Corp.
Amsco Solvent L-541	Toluene	Amsco Chemical Corp.
Barium Sulfate	same	J. T. Baker Chem. Co.
Cab-O-Sil	Fumed silica	Godfrey L. Cabot Co.
Calcium Octoate <u>5%</u>	Same	Tenneco Chemicals, Inc.
Calwhite	Ground calcium carbonate	Georgia Marble Co.
Cobalt Naphthenate <u>5%</u>	Same	Tenneco Chemicals, Inc.
D-2000	Amine terminated, difunctional polyoxypropylene	Jefferson Chemical Co.
DIAC #2	Amine salt	E. I. Dupont de Nemours
DMP-30	2,4,6-Dimethylmethylaminophenol	Rohm & Haas Co.
Dow #550 Oil	Silicone oil	Dow Corning Corp.
Epcar 346	Ethylene-propylene-diene monomer rubber	B. F. Goodrich Corp.
Epon 828	Bisphenol A epoxy (200 eq.wt.)	Shell Chemical Co.
FS-1265	Fluorosilicone	Dow Corning Corp.
GMF	Paraquinone dioxime	Uniroyal Inc.
Herclor C	Epichlorohydrin/polyoxypropylene copolymer rubber	Hercules, Inc.
Iron Yellow Y-03587	Iron oxide	C. K. Williams & Co.
Kadox 72	Zinc oxide	New Jersey Zinc Co.
Kaneka MS Polymer	Silane-terminated polyoxypropylene polymer	Kanegafuchi Chem. Co.
Lead Naphthenate <u>24%</u>	Same	Tenneco Chemicals, Inc.

TABLE VI

Lead Octoate <u>24%</u>	Same	Tenneco Chemicals, Inc.
Lead Peroxide	Same	Eagle-Pitcher Industries
Manganese Dioxide #808	Activated manganese dioxide	Shepherd Chemical Co.
Microtalc 3020	Talc	John K. Bice Co.
Neville 5722	Chlorinated paraffin	Neville Chemical Co.
Piperazine	Same	Aldrich Chemical Co.
PM Polymer	Mercaptan-terminated polyoxypropylene	Phillips Petroleum Corp.
PRC Permapol® Poly- siloxane Polymer	Proprietary cyanosilicone polymer	Products Research & Chemical Corp.
PRC Permapol® Poly- sulfide Polymer	Proprietary mercaptan-terminated polymer	Products Research & Chemical Corp.
PRC Primer #6	Proprietary silane-based primer	Products Research & Chemical Corp.
Regal 660R	Reinforcing carbon black	Godfrey L. Cabot Co.
Santicizer 278	Phthalate ester plasticizer	Monsanto Co.
Stan Clear TL	Dibutyltin dilaurate	Witco Chemical Corp.
Stannous Octoate	Same	Tenneco Chemical Co.
Sunpar 2280	Aliphatic oil	Sun Chemical Co.
Super-Ad-It	Diphenylmercury dodecylsuccinate	Tenneco Chemical Co.

XMSG DIA DROLS PROCESSING - LAST INPUT IGNORED

-- 1 OF 1 ***DTIC DOES NOT HAVE THIS ITEM***

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